



European Network on New Sensing Technologies for Air Pollution Control and Environmental Sustainability - *EuNetAir*

COST Action TD1105

**Microwave synthesis of nanooxides and their applications in microwave gas sensing**

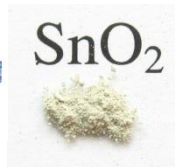
J Rossignol, B. De Fonseca , Pr D. Stuerger , Pr P Pribetich



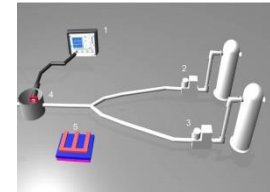
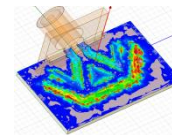
# Overview



## Synthesis of nanopowders



## Microwave gas sensing

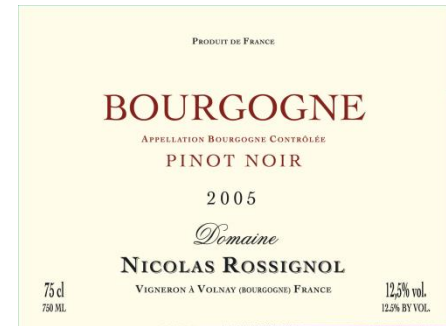
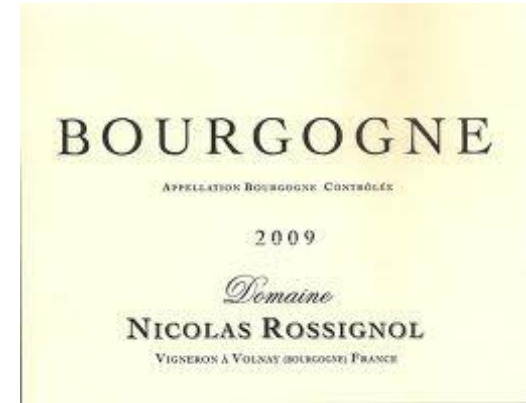
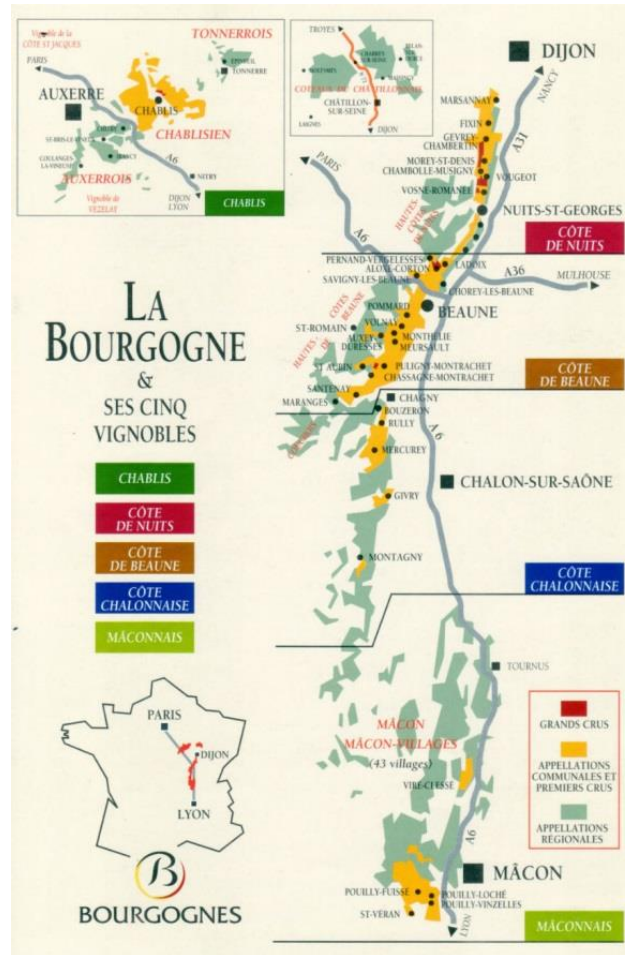


*challenge*

# Bourgogne



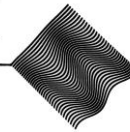
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# GERM



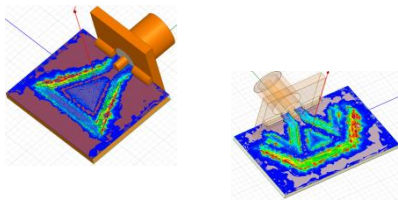
GERM



~~NA~~ AGORAS Technology

Modeling

Microwave field distribution



Design of microwave reactors

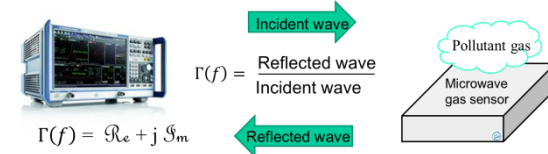
Microwave synthesis



$\text{SnO}_2$ ,  
 $\text{TiO}_2$ ,  
 $\text{ZrO}_2$ ,  
 $\text{Fe}_2\text{O}_3$ ...

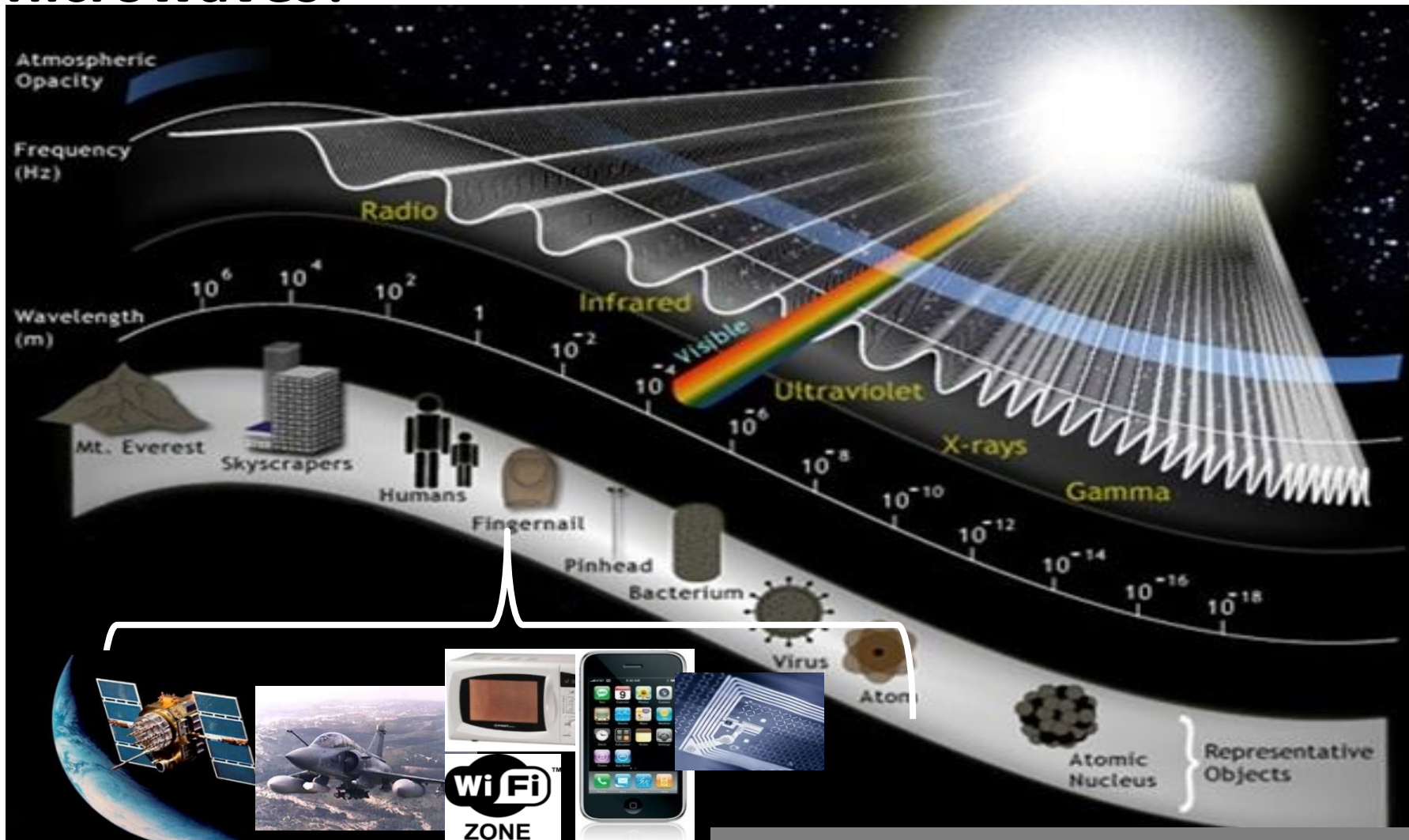
Microwave transduction

Liquid and gas sensing





# Microwaves?



Frequency: 300MHz to 300GHz  
 Wave length 1mm and 1m ( $\epsilon_r=1$ )

# Our Microwave synthesis

## The RAMO System

Core heating

Pressure control

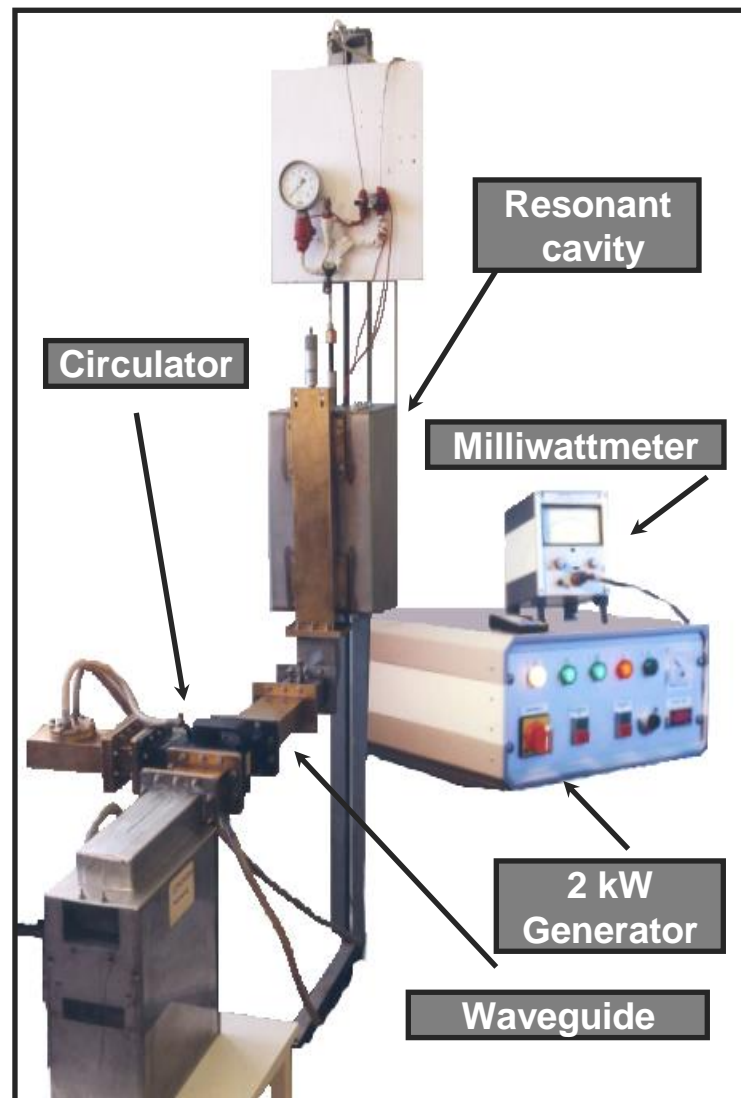
Temperature control

Atmosphere control

*In situ*  
measurements

**RATES :** temperature 5 to 15°C.s<sup>-1</sup>

pressure 1.2 MPa.min<sup>-1</sup>



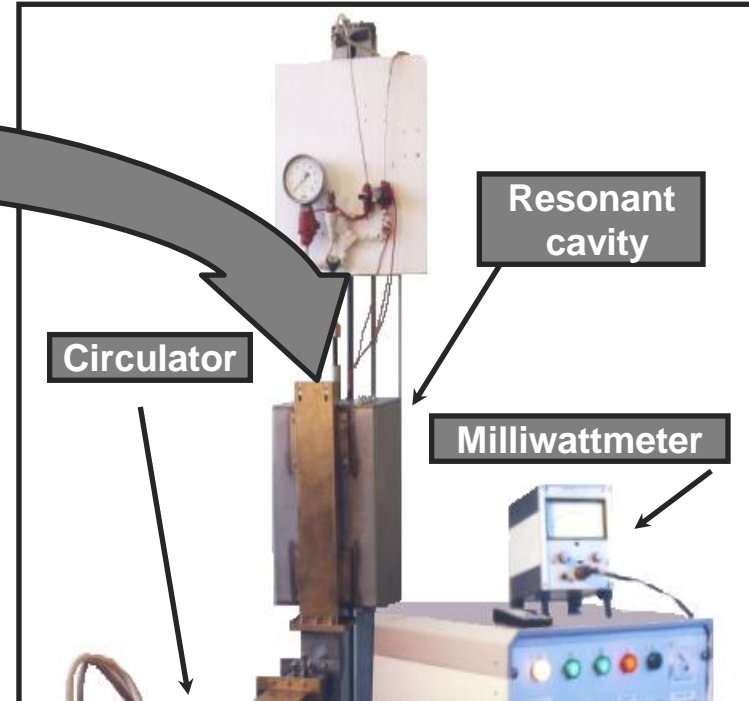
# Synthesis of nanopowders

The RAMO System

Oxide precursor, hydrochloric acid

Initial power 1 kW

Microwave heating duration  $\leq 60$  s



$\mu m$    $nm$

***Size BET confirmed by DRX***

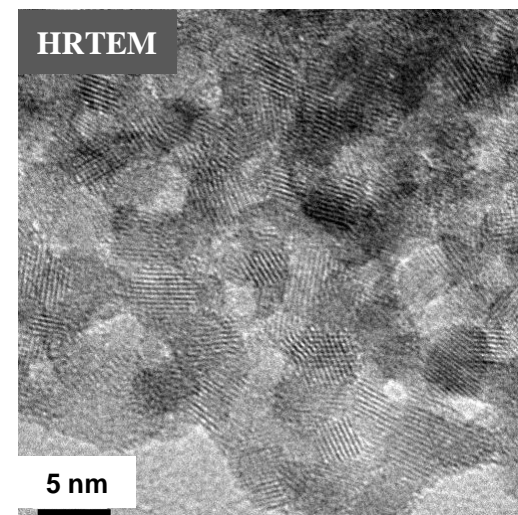
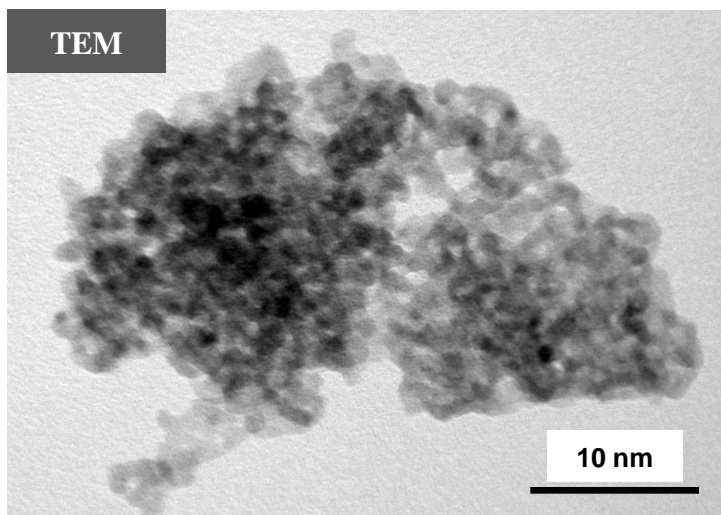
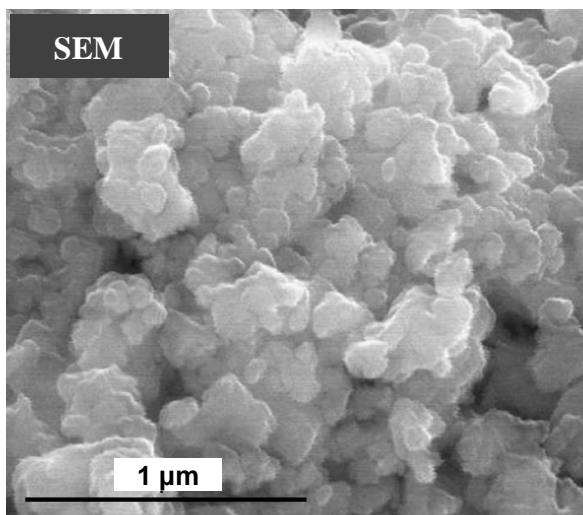
# SnO<sub>2</sub>

## Tin oxide (IV) by microwave thermohydrolysis (RAMO) :

SnCl<sub>4</sub> (Aldrich, 99,995%) + HCl (Prolabo, RP Normapur™)

DRX → Cassiterit (Fiche JCPDS 41-1445)

Microsonde XDE → Any trace of Cl



WIPO: WO/2009/050344, *Method for preparing nanoparticles of complex metal oxide*), with exclusive exploited licence to the society *Naxagoras Technology*.



# TiO<sub>2</sub>

Anatase

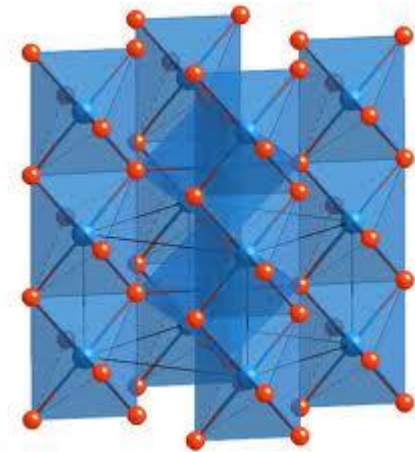


Rutile

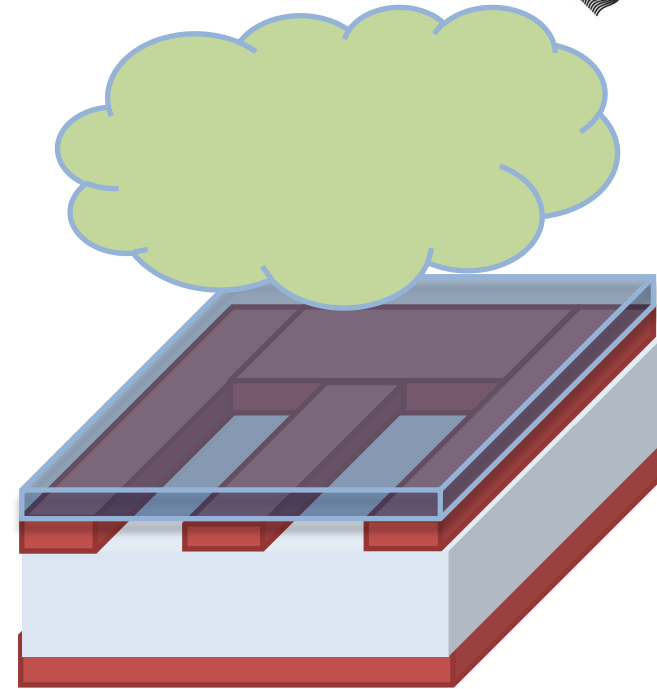
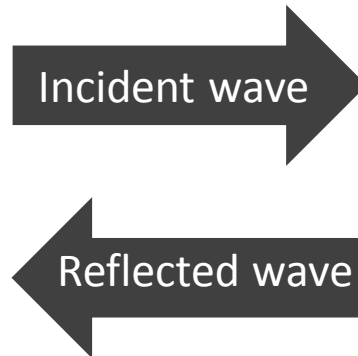


Difference of  
surface acidity

Effect of adsorption



# Microwave gas sensing



$$\Gamma(f) = \frac{\text{Reflected wave}}{\text{Incident wave}}$$

$$\Gamma(f) = \text{Re} + j \text{Im}$$

1 frequency  
1600 frequencies

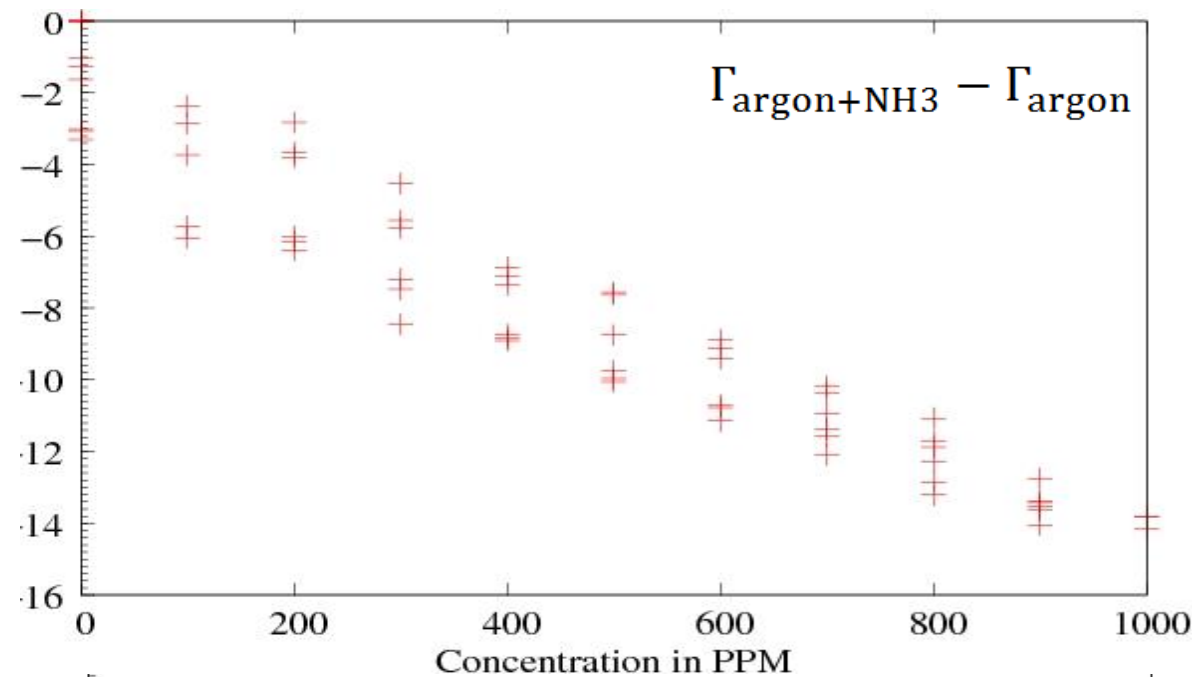
2 informations  
**a Signature**



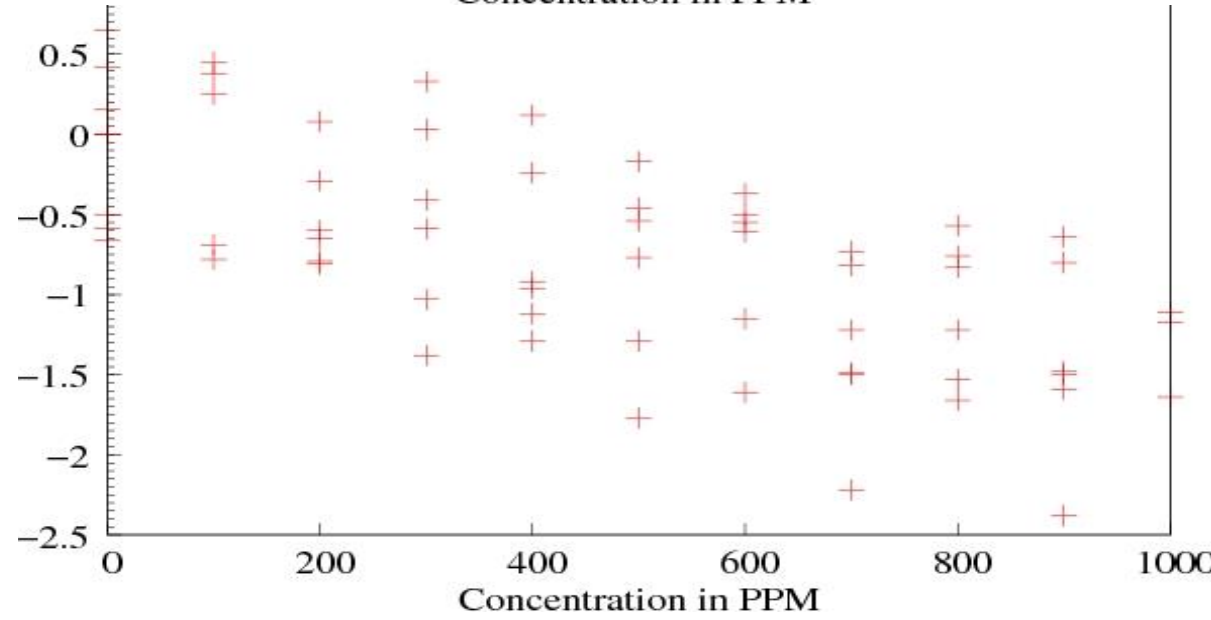
**SnO<sub>2</sub> , pollutant NH<sub>3</sub>**

$\Re(\Gamma)$   
 $10^{-4}$

$\epsilon \approx 25$

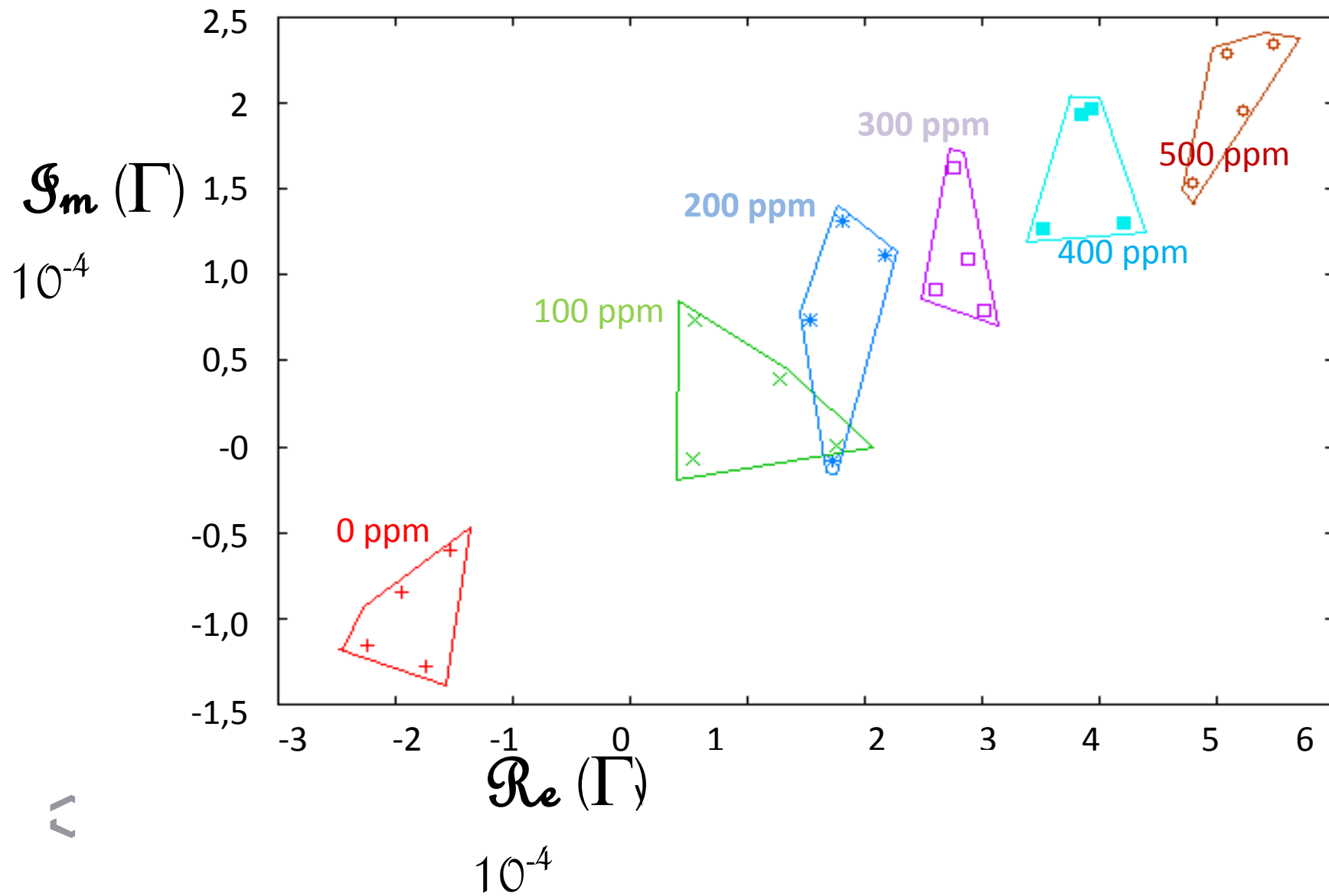


$\Im(\Gamma)$   
 $10^{-4}$



# TiO<sub>2</sub>, pollutant NH<sub>3</sub>

$\epsilon \approx 85$





# Conclusions and challenges

An innovative approach to gas sensing:

- Microwave synthesis of nano metal oxide
- Microwave gas sensing

➔ Future investigation:

- Impact of the size, the porosity and the specific surface area of the metal oxide on the reflected coefficient  $\Gamma$
- Effect of the temperature and humidity on the sensor's response
- Knowledge of the interaction phenomena and modeling of the sensor 's response.